

PATENT SPECIFICATION



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COMPLETE SPECIFICATION

NO DRAWINGS

High Temperature Lubricant

We, MOBIL OIL CORPORATION (formerly SOCONY MOBIL OIL COMPANY INC.) a Company organised under the laws of the State of New York, United States of America, of 5
150 East 42nd Street, New York 17, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed to 10 be particularly described in and by the following statement:—

This invention is directed to an improved lubricant composition for use in lubricating materials which must operate at high temperature for extended periods of time and is particularly directed to an improved lubricant for use on kiln chains.

In the manufacture of many products a final heating or drying step is involved. 20 Giant ovens have been developed for continuously conveying materials therethrough which are generally 300-1000 ft. (91-305 m.) in length and which utilize continuous chains travelling over sprocket wheels to convey 25 the articles through the heating zone. These kilns operate at approximately 400-800°F. (204-427°C.), depending upon the heat treating conditions required. The heat is supplied by heated air or gas which is circulated through the ovens by fans or blowers suitably located to maintain uniform temperature throughout the oven. 30 These ovens are obviously expensive and their continuous operation is essential or 35 extremely desirable to the manufacturing operations.

As a specific example of the problems involved, Gypsum Wall Board contains a substantial amount of moisture which is put 40 into the stucco during the manufacturing operation. These boards must be dried in order to harden the board prior to stacking and shipping. This drying operation is

accomplished in an oven or kiln 500 to 700 ft. (152 to 213 m.) long. The boards are carried through the kiln on roll conveyors at approximately seven feet (two meters) per minute. There are six to fourteen conveyors in each kiln stacked one above the other. Each individual roll of the conveyor is of the order of 4 or 5 feet (1.2 or 1.5 meters) long. The many rolls of the conveyor are mounted in bearings and a sprocket is fixed to one end. A chain engages and moves over the sprocket, rotating the rollers, which in turn slowly propel the wall board through the kiln. Of course, other designs are used, but they have in common operation of moving parts, generally with a drive chain, at the elevated oven temperature, providing an exceedingly difficult lubrication problem.

The chains are continuous and may extend outside the kiln or in some instances be contained completely within the kiln. If they extend outside the kiln, the extension is generally just sufficient to pass the kiln at the pull end and far enough on the back end to run over the return adjustment idlers. They are, therefore, about twice the length of the kiln in which they travel and nine-tenths to all of their 600-2000 ft. (182-610 m.) length is constantly within the kiln and exposed to the high kiln temperatures.

The turbulence created by the circulating hot air in the oven tends to carry in suspension fine particles of dust, dirt, dried stucco, board chips, fibers, and other particles from the product undergoing treatment which tend to deposit on the oil wet chain links, and rollers. The oil used to lubricate the mechanism, which previously has been a hydrocarbon oil containing a small amount of graphite, cracks at the high temperature of the oven, producing carbonaceous deposits on the chains. The particles become embedded in the carbonaceous material

[Pr]

which acts as a binder and these deposits seriously increase the chain load requiring additional power to move the chain. These deposits can become so serious that they cause the chain to get noisy and may even cause the chain to buckle and break.

Chain breakage and damage has been a serious problem in the gypsum wall board industry, the fiber glass industry and many other industries using these high temperature ovens or kilns. A variety of lubricants has been tested but none has been found completely satisfactory. It is customary, therefore, to permit the chain power requirement to rise to a critical level and then remove the chain for cleaning and scraping. This increased power requirement is, of course, expensive. The down time of the oven is expensive and inconvenient since it interrupts the normal flow of the product.

The present invention provides a lubricant for use, at elevated temperatures on oven conveyor chains comprising a water-in-oil emulsion, the water-phase content of said lubricant being 15 to 70% by weight, and the oil-phase content of said lubricant being 85 to 30% by weight, the oil phase comprising a hydrocarbon oil of 50 to 1000 SUS viscosity at 100°F (37.8°C) and, based on the weight of the emulsion, 0.1 to 5 percent by weight, sufficient to emulsify the water and oil, of an emulsifier, 0.1 to 5 percent by weight of a high-temperature stabilizer for said emulsion, the combined emulsifier and stabilizer having an HLB number of 2 to 8, and .05 to 5 percent by weight of a solid lubricant in finely divided form.

The invention further provides a lubricant for use at elevated temperatures on oven conveyor chains and the like which comprises a water-in-oil emulsion containing 25-45 percent by weight water, 0.25-2.00 percent by weight of oil-soluble calcium petroleum sulfonate (100% active) as an emulsifying agent, 0.25-3.0 percent by weight of metal and ammonium soaps of naphthenic acids having molecular weights of 315-500 as a stabilizer, the metal being sodium, potassium, lithium, calcium, strontium, or barium, the oil phase of said emulsion comprising a hydrocarbon oil of 50-400 SUS viscosity at 100°F (37.8°C) the ratio between the oil-soluble calcium petroleum sulfonate and the metal naphthenate being 5/95 to 95/5 by weight, and 0.5 percent by weight of colloidal graphite.

The lubricant disclosed hereinabove has proved to be suitable for use on equipment operating at elevated temperatures, particularly on chains and other members in dusty ovens operating for sustained periods of time at temperatures of 400-800°F. (207-427°C).

In accordance with one aspect of this

invention a water-in-oil emulsion is formed using an emulsifier suitable for forming such emulsion and using a high-temperature stabilizer to ensure stability of the emulsion at high temperatures of the order of 170°- 70 200°F (77-93°C). Dispersed in the water-in-oil emulsion is a finely divided solid lubricant, such as graphite, molybdenum disulfide, boron nitride, mica, vermiculite, talcum or tungsten disulfide. The emulsion formed by the emulsifier and stabilizer should have an HLB number of 2-8 and preferably 3-7. The emulsifier and stabilizer can be selected from nonionic, anionic or cationic surfactants.

The oil used is a hydrocarbon oil having a viscosity range of 50-1000 SUS at 100°F. (37.8°C). The preferred viscosity range, however, is 80-150 SUS particularly 100 SUS, at 100°F (37.8°C). The oil generally vaporizes and cracks during the lubrication of the oven conveyor chains and, hence, the Conradson Carbon Residue should be broadly less than 0.25% and preferably less than 0.05%. The oil phase of the emulsion is 85-30% by weight but is preferably 70-50% by weight. A particularly good lubricant formulation is obtained when using a white oil with the characteristics given hereinabove.

The emulsifier may be any one of a number of oil-soluble compounds broadly classified as anionic, nonionic, or cationic. In order to be effective emulsifying agents, they should be strongly oleophilic, i.e. 100 having an HLB of broadly 2-6 and preferably 3-5. Surfactants having an HLB below 2 while very strongly oleophilic tend to be poorer emulsifying agents while those above 6 tend to form O/W emulsions or so-called mixed emulsions being partly O/W and partly W/O and, therefore, unstable. Compounds of the anionic type having been found particularly useful are oleophilic alkaline earth petroleum sulfonates, oleophilic alkaline earth naphthalenic sulfonates, oleophilic alkaline earth C₁₂-C₁₈ alkyl phenates and potassium, sodium, ammonium, calcium, barium, strontium, lithium salts of acid phosphates, said phosphates having 110 been partially neutralized by esterification with C₆-C₁₂ alkyl phenols or ethylene oxide derivatives (2-12 mols ethylene oxide) of said phenols. Compounds of the nonionic type having shown utility are partial esters of 115 polyols and C₁₂-C₁₈ fatty acids, or partial esters of polyols and so-called wax acids, the latter produced by the oxidation of a mixture of petroleum hydrocarbons having a mean carbon chain length in the range of 125 C₂₀-C₃₅, such mixtures commonly being known as petrolatum or microcrystalline wax. Particularly good results have been obtained with partial esters of sorbitol and its dehydration product such as sorbitan 130

mono-oleate. These emulsifiers are used at a total concentration of 0.1-5.0 per cent by weight and preferably 0.3-3.0 per cent by weight.

5 This lubricant is designed for use on oven conveyor chains and the like operating at very high temperatures, such as 400-800°F. (204-427°C.). It has been found that a water-in-oil emulsion provides the best 10 lubrication for this purpose in that the water droplets are enclosed in oil and hence are able to remain in liquid form for some time period because of the protective oil cover. Furthermore, oil being the outer 15 phase contacts the hot metal to provide lubrication to the metal. The metal chain is so hot that the water flashes off as steam on contact thereby providing a substantial cooling effect and also a blasting effect which 20 effectively removes the hard layer of carbon on the chain. This carbon layer, unless removed, builds up to form a tight layer preventing the chain from bending easily and increasing the chain load substantially.

25 However, to be effective, the emulsion must be delivered to the hot chain in stable form, with the water particles substantially uniformly dispersed in the oil. In order to produce this result in this very hot environment, an emulsion which is exceedingly stable at elevated temperatures must be provided. The emulsion must remain stable at temperatures as high as 170-200°F. (77-93°C.) to be at all effective. High-temperature 30 stabilizers must, therefore, be used in the lubricant formulation. Suitable high-temperature stabilizers are surfactants having an HLB of broadly 8-16 and preferably 10-15. Among suitable agents of the 35 anionic class, metal soaps of naphthenic acids having molecular weights of 315-500 formed from ammonium and the metals sodium, potassium, lithium, calcium, strontium and barium have been found especially 40 efficacious. Also useful are calcium C₈-C₂₀ alkyl salicylate and C₈-C₁₈ alkyl phenols. Likewise useful are soaps of C₁₈-C₂₀ and preferably C₁₈-C₂₂ fatty acids from ammonium and the metals calcium, barium, lithium, 45 strontium, sodium and potassium. Also useful are salts of so-called wax acids or partial esters of wax acids and polyols, e.g. the ethanolamine salts or soaps of these acids or their partial esters. Useful stabilizers of the nonionic type are ethylene oxide derivatives (3-12 mols ethylene oxide) of C₈-C₁₈ alkyl phenols, ethylene oxide condensates (1-5 mols ethylene oxide) of C₁₈-C₂₄ fatty or rosin alcohols and ethylene oxide 50 condensates (4-20 mols ethylene oxide) of partial esters of fatty acids and polyols. Likewise useful are ethoxylated fatty acids and amides, such as oleyl or stearyl amides condensed with 3-8 mols of ethylene oxide, 55 the latter being mildly cationic in character.

Cationic stabilizers of merit are fatty amines (C₁₂-C₂₀) derived from oleic, palmitic and stearic acids and modified by condensation with 2-12 mols of ethylene oxide. Other suitable agents are copolymers of C₁₈-C₂₀ 60 α olefins and vinyl alcohol having a molecular weight of 4000-50,000, water soluble polyacrylamide having a molecular weight of 35,000-50,000, an oil-soluble partial ester of a polyhydric alcohol and a C₈ to C₂₂ fatty acid, and water soluble polyvinyl pyrrolidone having a molecular weight of 35,000-50,000. The stabilizers are used at a total concentration of 0.1-5.0 per cent by weight and preferably 0.3-3.0 per cent by weight.

The amount of high temperature stabilizer needed is based in part upon the operating condition. For chains operating at the lower end of the temperature range and where the length of time in the oven is minimum, less 65 stabilizer need be used. This is also influenced in part by the method of application of the lubricant to the chain. It is essential that the lubricant reach the chain in the form of a uniform emulsion so that the oil 70 phase will provide lubrication and the water droplets will provide the blasting effect desired.

The preferred emulsifiers are the oil-soluble sulfonates. The preferred materials 75 for making oil-soluble sulfonates are those obtained by sulfonation of mineral lubricating oil fractions which may be prepared by any of the well known and accepted methods in this art. Calcium petroleum sulfonate 80 may be used as the emulsifier and may be present in the blend in the amount of 0.1-5.0 per cent by weight of the total blend but preferably 0.3-3.0 per cent by weight can be used to provide entirely satisfactory results. 85 The calcium petroleum sulfonate, while primarily an emulsifying agent, supplies a certain amount of anti-corrosive action and anti-wear protection. It is preferable that the calcium petroleum sulfonate have a 90 molecular weight of at least 900. When the calcium petroleum sulfonate has a molecular weight of 1000 the emulsification is excellent. Particularly useful calcium sulfonates are the highly purified oil soluble 95 calcium petroleum sulfonates supplied by Sonneborn and Sons, Inc., which bear the trade names of Calcium Petronate HMW (High Molecular Weight) or Basic Calcium Petronate HMW.

It is found that the emulsion will rapidly deteriorate, especially under the influence of heat, when the calcium petroleum sulfonate is used alone and hence the mixture of calcium petroleum sulfonate and oil alone as 100 the oil phase of the lubricant for high temperature use is generally not satisfactory. As previously indicated, it is found necessary to add a stabilizer to the emulsion which will act to hold the emulsion together 105

at elevated temperatures. Unusually stable emulsions are found to occur when naphthenic acid soaps of sodium, potassium, ammonium, lithium, calcium, barium or strontium are used as the stabilizing medium. The molecular weight of the naphthenic acid is found to be critical, naphthenic acids of molecular weight less than 275 being found to possess little or no stabilizing action. Particularly useful are naphthenic acids of 275-1000 molecular weight. Outstandingly stable emulsions are obtained when using naphthenic acids identified as Sunaptic Acid "B" and Sunaptic Acid "C" (Registered Trade Marks), which are a group of saturated high molecular weight naphthenic acids, using sodium, potassium or lithium as the soap forming ingredient. The "B" acid has a molecular weight of 325, whereas the "C" acid has a molecular weight of 415. The "C" acid is somewhat better than the "B" acid, although both provide excellent results. Naphthenic acid identified as Sunaptic Acid "A" (Registered Trade Mark) having a molecular weight of 295, on the other hand, was found to provide fair but still usable stabilization of the emulsion. This lighter acid salt reached optimum stability at a lower concentration but this stability was inferior to the stability obtained with the heavier acid salt and was more critical than that obtained with the heavier acid salt. Salts of naphthenic acid of molecular weight 250, designated "D" however, were found to provide little or no benefit regardless of concentration, and regardless of whether the sodium, potassium, ammonium, lithium, calcium, strontium or barium salts were used. The preferred naphthenic acids are those having molecular weights of 315-500. The concentration of the stabilizing agent in the finished blend varies from 0.1-5.0 per cent by weight but preferably should be from 0.3-3.0 per cent by weight.

In order to ensure adequate lubrication of the chain and related parts in the hot oven and yet obtain the full cleaning effect of the gasification of the water, the water content of the emulsion must be 15-70 per cent by weight. A preferred water content is 30-50 per cent by weight of the water-in-oil emulsion. The water content in the emulsion has a blasting effect upon chain deposits, particularly when the chain is operated at the higher temperatures. As the emulsion hits the hot chain the water flashes to a gas, causing a rupture and flaking away of the carbonaceous deposits on the chain. This keeps the chain free and able to flex without strain. Since the water particles are surrounded by oil, however, the oil provides lubrication to the chain and its moving parts and protects the chain from detrimental contact with water at elevated

temperature.

It is essential for proper lubrication of these hot oven chains to have dispersed in the emulsion lubricant a solid lubricant such as graphite or molybdenum disulfide or tungsten disulfide or boron nitride or mica or vermiculite or talc which is dispersed in the oil phase and remains on the chain after the intense heat has altered or driven off the remainder of the lubricant. Of the solid lubricants mentioned, the preferred solid lubricants are graphite, molybdenum disulfide and tungsten disulfide, with the most preferred solid lubricant being graphite. The solid lubricant can be dispersed in an oil or distillate hydrocarbon in concentrated form for admixture with the emulsion or it can be distributed directly with the oil by well known procedures. The graphite or other solid lubricant may or may not be mixed with a satisfactory graphite dispersant depending upon mixing procedure, type of emulsion selected viz. concentrated or dilute, and the decision to use a graphite dispersant may also depend in part upon the conditions under which the water-in-oil emulsion will be used and the oven operating conditions. Skill in blending and using graphite containing water-in-oil emulsions can be rapidly acquired, bearing in mind that a deposit of the graphite must be laid down on the hot chain to ensure adequate lubrication and the graphite dispersant must be compatible with the emulsifier system. The dispersed solid lubricant, such as graphite, should be added in the amount of 0.05-5% and preferably 0.1-2% by weight of the final composition. The graphite may be supplied first as a 1 part in ten dispersion in light oil or naphtha. This dispersion can then be readily mixed with the remainder of the emulsion lubricant to provide the finished blend. The particle size of the solid lubricant should be broadly 0.25 to 50 microns but preferably 0.5 to 5 microns.

In the following Examples the combined emulsifier and stabilizer have an HLB number of 2 to 8, the HLB number being obtained by multiplying the percentage by weight of each component of the emulsifier/stabilizer system by its HLB number and adding the products.

Example I

A conventional prior art oven conveyor lubricant was used consisting of 37.9% by volume of a paraffin oil having a viscosity of 60 SUS at 100°F. (37.8°C.), 56.7% by volume of a naphthenic oil having a viscosity of 100-110 SUS at 100°F. (37.8°C.) and 5.2% by volume of a colloidal graphite dispersion containing 1 part of graphite in 9 parts of mineral spirits (particle size about 2 microns). This lubricant, when used on oven conveyor chains operating at tempera-

tures over about 400°F. (204°C.) used in the manufacture of fiber glass, produced a very hard carbon formation on the chain which ultimately caused the links of the chain to freeze. Some of the links would then wear excessively so that flat spots occurred in the chain. Furthermore power consumption greatly increased after the hard carbon formation occurred, making it necessary to remove the chains for cleaning and replacement of worn links. This is an extremely expensive proposition amounting to as much as \$10,000 per year per chain.

Example 2

15 A fine stable water-in-oil emulsion lubricant for oven conveyor chains can be formed soluble calcium petroleum sulfonate as the basic emulsifier, 41.5 per cent by weight of water, 0.8 per cent by weight potassium naphthenate (using naphthenic acid of 325 M.W.) as the stabilizer, 0.5 per cent by weight colloidal graphite and the balance paraffin oil having a viscosity of about 100 SUS at 100°F. (37.8°C.). The mixture is 20 emulsified by a well known method and is then ready for use. As a test of stability, a sample of this emulsion was placed in a tall 4 oz. (113.4 gm.) oil sample bottle up to a level of 130 mm. and subjected to a seven 25 day storage test at 170°F. (77°C.). The water separated was nil and the oil separated was about 7 mm. This lubricant, when applied to high-temperature oven conveyor chains, does a superior lubricating job when compared to the prior art lubricant of Example 1.

Example 3

Another fine stable water-in-oil emulsion lubricant for oven conveyor chains was 40 formed in the following manner:

Two-thirds of the total amount of oil to be used, i.e. 52.05% by weight, was charged to a steam-heated kettle equipped with mechanical agitation. The oil used was a medicinal white oil having a viscosity of 45 about 110 SUS at 100°F. (37.8°C.). To this oil were added 0.19% by weight of naphthenic acid (M.W. 295), 0.19% by weight of naphthenic acid (M.W. 415), and 0.10% by 50 weight of hydrogenated marine oil fatty acids. The mixture was heated to 140°F. to 150°F. (60°C. to 66°C.) and 0.10% by weight of glacial acetic acid was added, followed by 0.39% by weight of caustic 55 potash solution (45% by weight active material), the blend was stirred for an additional 15 minutes whereupon the temperature was raised to 190°F. (88°C.). At this point 2.37% by weight of basic calcium 60 petroleum sulfonate (970-1000 M.W.; 40-45% active) as added and the temperature raised to 260°F. (127°C.). This temperature was maintained for 5-10 minutes and the batch quenched with the remaining one-third of the 65 mineral oil. While adjusting the batch

temperature to 175-185°F. (79.5-85°C.), 0.48% by weight of a mixture of octylated diphenyl amines was added. 39.83% by weight of water was heated in a separate kettle and added to the oil phase over a period of 15-30 minutes with vigorous agitation. After homogenizing to ensure small particle size of the water droplets, the emulsion was cooled and 4.30% by weight of colloidal graphite dispersion (1 part 75 graphite in 9 parts of mineral spirits) was added. The resultant preparation had excellent stability at temperatures of 170-200°F. (77-93°C.).

Example 4

A portion of the lubricant of Example 3 was applied to a commercial oven chain used in the curing of fiber glass. This oven measured approximately 95 ft. (29 m.) in length, 10 ft. (3 m.) in width, and 10 ft. (3 m.) in height. The oven contained two conveyor flights—upper and lower—extending almost the full length of the oven. The lower flight was fixed but the upper flight was adjustable in height to provide a means of controlling the thickness of the mat. The sprockets were located within the oven and at each end of the oven. The sprockets measured about 3 ft. (0.9 m.) in diameter and were about 90 ft. (27 m.) apart, giving a total chain length of about 200 ft. (61 m.). Since four chains were located in this oven the total chain length measured about 800 ft. (244 m.). The oven conveyors were driven by an Oilgear hydraulic unit, the hydraulic pressure varying from 400-1200 p.s.i. (28.1-84.3 kg/cm²) (guage) according to the load factors. Given a set operating condition, the only variable was the effectiveness of the lubrication or the carbon build-up on the chain. The 400 p.s.i. (28.1 kg/cm²) is roughly equivalent to 60 horsepower (44.7 KW) whereas the 1200 p.s.i. (84.3 kg/cm²) is roughly equivalent to 120 horsepower (89.5 KW) requirement. The oven was maintained at about 400-600°F. (204-316°C.) during the test operation.

The test lubricant was pumped intermittently and discharged under pressure onto the link of the chain in an amount to supply sufficient lubricant. Before the test lubricant was applied the chains had been lubricated with the oil of Example 1 and a heavy deposit of carbonaceous material mixed with fiber glass had built up on the chain. When the test lubricant was substituted the deposits on the chain commenced to decrease. The hydraulic pressure of the drive unit gradually reduced from over 1000 p.s.i. (70.3 kg/cm²), to 700-800 p.s.i. (49.2-56.2 kg/cm²) at the same general operating conditions.

Example 5

In two other installations using conven-

tion prior lubricant similar to the lubricant of Example 1, hydraulic pressure had built up gradually until it reached relief pressure over about 1100 p.s.i. (77 kg/cm²) (guage), 5 and the unit automatically shut down. Upon changing to the lubricant shown in Example 3, this pressure was reduced to 500 p.s.i. (35.2 kg/cm²) (guage) over a period of less than two weeks and subsequently decreased 10 to 400 p.s.i. (28.1 kg/cm²) (guage), indicating that horsepower (kW) requirements were reduced by almost 50 per cent.

Example 6

A test of the lubricant of Example 1 without the graphite was made on the oven chains described in Example 3. The lubrication of the chain was found to be inferior and the chain commenced to squeal from inadequate lubrication. This test had to be discontinued to prevent damage to the equipment from lack of lubrication.

It has been estimated that the use of the water-in-oil lubricant of this invention will prolong the active life of these high temperature oven conveyor chains from about one year to about two years. Since the chains are expensive and down time is expensive, the economic advantage of using this lubricant is measured in many thousands of 20 dollars in savings to the user.

Example 7

A suitable oven conveyor lubricant is obtained by mixing 0.5% by weight basic calcium salicylate, 0.5% by weight basic 35 calcium petroleum sulfonate, about 35% by weight water, 0.5% by weight dibenzyl disulfide, 0.5% by weight glycerol monoleate, 0.5% by weight colloidal graphite and 62.5% by weight of a naphthenic petroleum 40 oil having a viscosity of about 200 SUS at 100°F. (37.8°C.). The materials are mixed and emulsified by methods known in the art and a satisfactory stable emulsion is formed which remains stable at elevated temperatures.

Example 8 ...

A suitable oven conveyor lubricant for high temperature operation is obtained by mixing 1.5 per cent by weight lithium naphthalene (using a naphthenic acid having a molecular weight of 415), 0.5% by weight of calcium petroleum sulfonate (about 1000 molecular weight), 60% by weight of petroleum white oil, 5% by weight of colloidal 55 graphite in naphtha (1/10th or 0.5% by weight graphite), and 33% by weight of water. The ingredients are emulsified by procedures well known in this art to yield a high stable water-in-oil emulsion lubricant 60 suitable for high temperature duty and oven conveyor chains.

Example 9

An oil-in-water emulsion lubricant containing 1 part oil and 3 parts water (using 65 a conventional oil-in-water emulsifier

system) was combined with 0.5% by weight of the final formulation of colloidal graphite and tested on the commercial oven disclosed in Example 3. It was soon noted that the power requirement increased, indicating inadequate lubrication and the formation of hard carbon on the chains. The chains began to squeal noticeably. After several days operation the power requirement had increased to such an extent that 70 the test was discontinued to prevent damage to the chains. Reuse of the lubricant of Example 2 stopped the squealing of the chain and brought the power requirement back to the level prevailing before this test 75 was commenced.

Example 10

An oil-in-water emulsion lubricant containing equal parts of oil and water (using a conventional oil-in-water emulsifier 85 system) was combined with 0.5% by weight of the final formulation of colloidal graphite and tested on the commercial oven disclosed in Example 3. The results were very 90 similar to those disclosed in Example 9.

Example 11

A fine stable water-in-oil emulsion lubricant was formed by mixing 2.36% by weight of basic calcium petroleum sulfonate (970-1000 M.W.; 40-45% active), 0.95% by 95 weight of naphthenic acid (M.W. 415), and 0.58% by weight of caustic potash solution (20% active) with approximately one-third of a total of 51-77% by weight of solvent refined paraffin (100 SUS at 100°F. [37.8°C.]). This mixture was heated to 150°F. (66°C.) and the remaining two-thirds of the base oil was added. Subsequently, 0.42% by weight of a mixture of octylated diphenyl amines and 4.72% by weight of 105 colloidal graphite dispersion (one part graphite in nine parts mineral spirits) were blended into the mixture. Finally 39.20% by weight of water previously heated to 150°F. (66°C.) was added with rapid agitation. A sample of the resulting fine particle emulsion was stored at 170°F. (77°C.). Examination after five days showed no water separation and only 2.5% of free oil. The same sample after eleven days still 115 showed no separation of water and only 5.0% of supernatant oil. It will be appreciated that in water-in-oil emulsions separation of oil is a minor deficiency since it can easily be mixed with the remaining 120 emulsion by mild agitation inasmuch as the oil constitutes the continuous or outer phase. Another sample of the above-described oven conveyor lubricant was stored at a temperature close to the boiling point of water, i.e. 125 at 200°F. (93°C.). This sample, which was stored under such severe conditions, was examined at similar time intervals (5 days; 11 days) and after five days still exhibited no free water and only 3% of free oil; after 130

a total of eleven days of exposure water separation amounted to about 1.5% and oil separation to about 8%. The outstanding heat stability of this material makes it of particular utility for use as a high-temperature, oven-conveyor lubricant.

Example 12

A fine stable high-temperature oven-conveyor lubricant is formed by mixing 1.8% by weight of surbitan mono-ooleate, (HLB=4.5) 1.2% by weight of polyoxyethylene sorbiton trioleate (20 mols of ethylene oxide) (HLB=11), 52.0% by weight of solvent refined naphthenic petroleum oil of a viscosity of 100 SUS at 100°F. (37.8°C.), 3-4 p.p.m. of a defoamant (Dow Corning Fluid 200-12,500 centipoises, which is a synthetic silicon product supplied by Dow Chemical bearing a trademark not registered with the United States Patent Office), 5% by weight of colloidal graphite (one part graphite to nine parts mineral spirits) and 40% by weight of water. This lubricant was stored for 40 hours at 170°F. (77°C.) and showed no water separation and only 2% oil separation. This lubricant is an excellent lubricant for hot oven conveyor chains (operating at 400-800°F. [204-427°C.]). The HLB of the combined emulsifier and stabilizer is $60\% \times 4.5 + 40\% \times 11 = 2.7 + 4.4 = 7.1$.

Example 13

Another fine stable high-temperature oven-conveyor lubricant was formed by mixing 2.5% by weight of surbitan mono-ooleate, 0.5% by weight of the condensation product of oleyl amide and 5 mols of ethylene oxide, 47% by weight of a solvent-refined naphthenic petroleum oil having a viscosity of 100 SUS at 100°F. (37.8°C.), 0.1% by weight of a defoamant as described above (Dow Corning 200 Fluid, 1000 centipoises, 10% solution in kerosine), 49% by weight of water and 1% by weight of molybdenum disulfide (96% of total number of particles below 2 microns in size). This lubricant was stored at 170°F. (77°C.) for 24 hours and showed no water separation and only 3% oil separation. This formulation is an excellent conveyor chain lubricant for chains operating at temperatures in the range of 400-800°F. (204-427°C.).

The test programme conducted has demonstrated that oven conveyor chains operating at temperatures over 300°F. (149°C.) and more particularly at temperatures of 400-800°F. (204-427°C.) require for maximum performance a water-in-oil emulsion in which a stabilizer is used with the base emulsifier to provide a stable emulsion at temperatures as high as 170-200°F (77-93°C.). This programme has shown that a solid lubricant, such as colloidal graphite, molybdenum disulfide, tungsten disulfide or boron nitride, must be dispersed in the

emulsion to provide adequate lubrication of the chains. The lubricant, for most efficient lubrication, should be provided either intermittently, or continuously to the chain without a long dwell period in the oven. 70

WHAT WE CLAIM IS:

1. A lubricant for use at elevated temperatures on oven conveyor chains and the like comprising a water-in-oil emulsion, the water-phase content of said lubricant being 75 15 to 70% by weight, and the oil-phase content of said lubricant being 85 to 30% by weight, the oil-phase comprising a hydrocarbon oil of 50 to 1000 SUS viscosity at 100°F (37.8°C.) and, based on the weight of the emulsion, 0.1 to 5 percent by weight, sufficient to emulsify the water and oil, of an emulsifier, 0.1 to 5 percent by weight of a high-temperature stabilizer for said emulsion, the combined emulsifier and stabilizer having an HLB number of 2 to 8, and .05 to 5 percent by weight of a solid lubricant in finely divided form. 80

2. A lubricant according to Claim 1 comprising 0.3 to 3 percent by weight of said 90 emulsifier.

3. A lubricant according to Claim 1 or Claim 2 comprising 0.3 to 3 percent by weight of said stabilizer.

4. A lubricant according to any one of 95 Claims 1 to 3 wherein the combined emulsifier and stabilizer have an HLB number of 3 to 7.

5. A lubricant according to any one of 100 Claims 1 to 4 wherein said hydrocarbon oil has a viscosity of 80 to 150 SUS at 100°F. (37.8°C.).

6. A lubricant according to any one of 105 Claims 1 to 5 wherein said oil has a viscosity of 100 SUS at 100°F (37.8°C.).

7. A lubricant according to any one of 110 Claims 1 to 6 wherein said hydrocarbon oil has a Conradson Carbon Residue of less than 0.05 percent.

8. A lubricant according to any one of 115 Claims 1 to 7 wherein the amount of the oil-phase is from 70 to 50 percent by weight and the water content of said lubricant is from 30 to 50 percent by weight.

9. A lubricant according to any one of 120 Claims 1 to 8 comprising 0.1 to 2 percent by weight of said solid lubricant.

10. A lubricant according to any one of 125 Claims 1 to 9 wherein said emulsifier is an oleophilic alkaline earth petroleum sulfonate, an oleophilic alkaline earth naphthalene sulfonate, an oleophilic alkaline earth C₁ to C₁₈ alkyl phenate, a potassium, sodium, ammonium, calcium, strontium, barium, or lithium salt of an acid phosphate, said phosphate having been partially neutralized by esterification with a C₈ to C₂₁ alkyl phenol or an ethylene oxide derivative (2 to 12 mols ethylene oxide) of said phenol, a partial ester of a polyhydric alcohol and a C₁₀ 130

- to C₂₁ fatty acid, or a partial ester of a polyol and a wax acid.
11. A lubricant according to any one of Claims 1 to 10 wherein said emulsifier is sorbitan mono-oleate.
12. A lubricant according to any one of Claims 1 to 11 wherein said stabilizer is a soap of naphthenic acid having a molecular weight of 315 to 500 formed from sodium, potassium, ammonium, lithium, calcium, strontium or barium; calcium C₈ to C₂₀ alkyl salicylate, C₄ to C₁₈ alkyl phenol, a copolymer of a C₁₀ to C₁₈ α -olefin and vinyl alcohol having a molecular weight of 4,000 to 50,000, a water-soluble polyacrylamide having a molecular weight of 35,000 to 50,000, water-soluble polyvinyl pyrrolidone having a molecular weight of 35,000 to 50,000, an oil-soluble partial ester of a polyhydric alcohol and a C₄ to C₂₀ fatty acid, a soap of a C₁₆ to C₂₀, and preferably C₁₈ to C₂₀, fatty acid formed from potassium, sodium, ammonium, lithium, calcium, strontium or barium, an ethylene oxide condensate (1 to 5 mols of ethylene oxide) of a C₁₀ to C₂₁ fatty or rosin alcohol, or a partial ester of a polyhydric alcohol and a C₁₀ to C₂₁ fatty acid, said ester being condensed with 4 to 20 mols of ethylene oxide.
13. A lubricant according to any one of Claims 1 to 12 wherein said solid lubricant is graphite, molybdenum disulfide, boron nitride, mica, vermiculite, talcum or tungsten disulfide.
14. A lubricant according to any one of Claims 1 to 13 wherein said hydrocarbon oil is a white oil.
15. A lubricant for use at elevated temperatures on oven conveyor chains and the like which comprises a water-in-oil emulsion containing 25 to 45 percent by weight water, 0.25 to 2.00 percent by weight of oil-soluble calcium petroleum sulfonate (100% active) as an emulsifying agent, 0.25 to 3.0 percent by weight of ammonium or metal soaps of naphthenic acids having molecular weights of 315 to 500 as a stabilizer, the metal being sodium, potassium, lithium, calcium, strontium, or barium, the oil phase of said emulsion comprising a hydrocarbon oil of 50 to 400 SUS viscosity at 100°F. (37.8°C.), the ratio between the oil-soluble calcium petroleum sulfonate and the metal naphthenate being 5/95 to 95/5 by weight, and 0.5 percent by weight of colloidal graphite.
16. A lubricant in accordance with Claim 1 substantially as hereinbefore described with reference to any of the Examples.

For the Applicants.

CARPMAELS & RANSFORD.

Chartered Patent Agents.

24. Southampton Buildings,

Chancery Lane,

London, W.C.2.

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